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PROTECTIVE COATINGS FOR THERMOCOUPLE SHEATHS

(Title Unclassified)

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P. J. Levine
Materials Department
May 24, 1965

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MAY 28 1965

• Mr. J. L. Dooling
NERVA Subcontract Manager
Rocket Engine Operations - Nuclear
Department 7140
Aerojet-General Corporation
Post Office Box 1947
Sacramento, California

Subject: WANL-TME-1164, "Protective Coatings for Thermocouple Sheaths",
dated May 24, 1965

Dear Mr. Dooling:

Transmitted herewith are five (5) copies of the subject report. This report discusses the capability of vapor-deposited tungsten coatings and surface-carburized layers to protect thermocouple sheaths from destruction in a high-temperature carbon and hydrogen environment.

Respectfully,

Original Signed By

H. F. Faught

H. F. Faught *clw*

Program Manager

NERVA Nuclear Subsystem

Enclosures (5)

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PROTECTIVE COATINGS FOR THERMOCOUPLE SHEATHS
(Title Unclassified)

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Materials Engineering and Specifications

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I ABSTRACT

A protective coating on refractory metal sheaths is required for reactor thermocouples intended to operate in a hydrogen-carbon environment at 5000°R for one hour. Both tungsten vapor-deposited coatings and surface carburization of the sheath appear to be satisfactory means of preventing hydriding and growth of tantalum. For the use of vapor-deposited tungsten, Vendor B is capable of supplying a high-quality product superior to that of Vendor A. The described tests on molybdenum and tungsten-26% rhenium will determine the capability of vapor-deposited tungsten coatings to prevent metal-carbon eutectic reactions from occurring at the maximum temperatures of operation.

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II INTRODUCTION

The measurement of temperatures to 5000°R (2500°C) in the carbon and hydrogen environment of the NERVA reactor requires the use of high-temperature materials, such as the refractory metals and oxides. Unfortunately, some of the available materials are not completely stable in the environment and, as a result, must be protected by the more stable ones. The thermocouples which incorporate protective coatings on the sheaths are those for Stations 26, 32, and 45. The Station 26 and 32 couples are composed of tungsten and tungsten-26% rhenium wires with beryllia insulators and tungsten-coated molybdenum sheaths. The tungsten coating is specified to protect the molybdenum from direct contact with carbon and the possible resultant formation of the molybdenum-carbon eutectic composition at 4470°R (2210°C),^{1, 2} which is the maximum temperature the Station 32 couple is expected to measure.³ Although the Station 26 thermocouples will not see this high temperature, the tungsten coating is applied to add reliability to the sheath, since these couples are used for control purposes as well as diagnostic measurements.

The Station 45, high-temperature thermocouple is composed of tungsten and tungsten-26% rhenium wires with beryllia insulators and a dual-sheath arrangement of molybdenum covered by tungsten-coated tantalum. The maximum temperature for this couple is nearly 5000°R (2500°C),³ which points out the reason for covering the inner molybdenum sheath with a tantalum tube to prevent the molybdenum-carbon eutectic formation. However, tantalum absorbs hydrogen at a high rate which results in the formation of hydrides and subsequent embrittlement of the sheath. Hydriding occurs at temperatures up to 2650°R (1200°C) with the most severe range being 1930°R (800°C) to 2290°R (1000°C).⁴ Because of this, the tantalum tube is coated with a layer of tungsten by vapor deposition to inhibit hydrogen penetration and prevent brittle failure of the sheath. The quality and capability of the tungsten coating are being determined. Also, a pre-carburized surface on the tantalum is being evaluated as a possible means of protection from hydrogen.

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III TUNGSTEN COATING EVALUATIONS

The tungsten coating used for thermocouple protection is applied by vapor deposition from gaseous tungsten-hexafluoride (WF_6). Long lengths of tubing are zone heated with an induction coil while the WF_6 is chemically reduced by hydrogen to deposit tungsten on the hot substrate surface. Careful preparation of the tube surface and close control of the temperature, pressure, and movement of the hot zone regulate the rate of deposition and final quality and adherence of the coating. This process results in a uniform and continuous coating with a columnar grain structure and a nominal surface roughness of 20 microinches. A typical example of this coating is shown in Figure 1.

Original thermocouple specifications permitted a flame-sprayed tungsten coating as an alternate, but this has since been eliminated. The flame-spray process produces a porous, discontinuous, and non-uniform coating, shown in Figure 2, which does not afford the required protection for the substrate material. The primary reason for the contrast in the quality of the two processes is the method by which each is applied. In flame spraying, particles, often contaminated with oxides, are sprayed onto the substrate to which they adhere by mechanical bond or, if conditions of heating are just right, by slight interdiffusion. Vapor deposition is an atom-by-atom growth which results in a uniformly deposited coating. The "line-of-site" characteristic of flame spraying destroys the uniformity of thickness required for coatings.

The deposition of a high-quality tungsten coating on molybdenum is relatively easy to obtain according to vendors. The sample shown in Figure 1, which was coated by the WANL Materials Department, demonstrates the excellent adherence that can be achieved on molybdenum with diffusion across the interface.

However, tungsten is not so easily deposited on tantalum. Since the thermal expansion of Ta is almost twice that of W, cooling from the deposition temperature (550°C) to ambient conditions causes the Ta to contract more than the W, tending to destroy adherence. Although

not presently used, a post-deposition heat treatment before cooling might cause enough interdiffusion of the two materials to prevent separations like that shown in Figure 3. An additional deterrent to a good bond is the preferential reduction of WF_6 by Ta, which results in the formation of tantalum tri-fluoride on the surface of the tube.⁵ Entrapment of this fluoride beneath the W will prevent suitable adherence of the coating. By carefully selecting the deposition parameters, including the H_2/WF_6 flow ratio, the formation of the volatile tantalum penta-fluoride can be forced to prevent contamination of the substrate surface.

The high-temperature thermocouple presently has a nominal 0.004-inch thick vapor-deposited tungsten coating on the sheath. The prime supplier of the coating (Vendor A) has not been able to produce a continuous, adherent, and uniformly thick layer of tungsten on tantalum. Typical examples of his product are shown in Figures 4 and 5. The former shows the columnar structure with a radial crack in a grain boundary and a separation at the interface between the coating and tube surface. This type of inferior coating permits hydrogen to get through the W layer and spread over the tantalum surface causing hydriding and brittle failure of the sheath like that shown in Figures 6 and 7. The transverse section in Figure 5, when compared to that in Figure 4, shows about a 2:1 variation in coating thickness on diametrically opposite sides of the tube (note difference in magnification).

Because of the apparent inability of Vendor A to satisfactorily coat tantalum tubes with tungsten, a second source with the capability to vapor-deposit refractory metal coatings on 4-foot-long tubes was evaluated. Several tantalum sheaths were coated by this source (Vendor B) and examined metallographically before and after testing, as shown in Figures 8 through 13. Figure 8 is a representative example of the quality of tungsten coating applied by Vendor B. The uniformity and continuity are superior to Vendor A's and the adherence to the tantalum is excellent. Ordinarily the vapor deposited coating is applied in one pass for the required thickness, but the process can be stopped and restarted. The resultant coating would appear like Figure 9 with a laminated-type structure of two or more layers. The effects of this structure on the integrity of the coating have not been established, but it is suspected of being a weaker condition.

An additional problem experienced in the vapor-deposition process is the formation of nodules, which are most likely nucleated by foreign particles on the surface or localized high temperatures caused by arcing of the induction coil. Macroscopic and microscopic views of these defects are shown in Figures 10 and 11, respectively. The grains in the nodule grow radially from the nucleus, often leaving a void in the center. Figure 12 shows the start of a nodule on the initial coating pass that has been stopped in growth by a second deposition of tungsten. The nodules ruin dimensional tolerances if they are not ground smooth. Nodular structures are not characteristic of coatings processed by Vendor B but are shown merely because of their presence in one of the first tubes that were evaluated. Grinding marks have appeared on the surface of as-received thermocouples coated by both vendors indicating the possible presence of nodules.

Figure 13 illustrates the post-test conditions of a coated sheath from Vendor B that was subjected to a carbon and hydrogen environment at high temperature. The quality of the coating was maintained and it afforded adequate protection for the substrate tantalum, which is relatively unharmed as compared to Figure 7. Recent thermocouple qualification tests were performed in which one sheath coated by each vendor was represented. Figure 14 demonstrates the superior capability of Vendor B's coating to protect the tantalum from hydriding in the region most susceptible to hydrogen embrittlement. Both couples were tested in the same graphite element at the same time. After test, the couple coated by Vendor B was easily extracted from the element in this region, but the other had to be chipped out of the channel, leaving the fragments of tungsten and tantalum shown in the photo. The tungsten did not protect the sheath from hydriding, which caused the tantalum to swell and crack the coating, thus binding the couple in the element.

Only recently have reactor thermocouples been ordered with a tungsten coating on tantalum provided by Vendor B. Unfortunately, these sheaths were contaminated on the inside surface during the vapor deposition process, leaving a residue of tantalum fluoride and tantalum di-oxy-fluoride. During high-temperature qualification tests on these thermocouples, the tantalum compounds reacted causing severe damage to the sheaths and elements.

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Because of this, tantalum has been eliminated as a sheath material in NRX-A4.⁶ However, the couple of cases where high-quality tungsten coatings from Vendor B have adequately protected the tantalum, i.e., Figure 14, prove the capability of this means of protection. Additional and more detailed testing will warrant the reinstatement of tungsten coated tantalum sheaths on reactor high-temperature thermocouples.

IV TUNGSTEN COATING VS. TANTALUM CARBIDE

Two possibilities have been considered as protective coatings on tantalum for the prevention of hydrogen embrittlement. They are: 1) vapor deposition of a 0.004-inch layer of tungsten, and 2) carburization of the surface of the tantalum tube. Tungsten was selected for the prime design because the parameters and equipment set-up had been determined and proven satisfactory. The latter choice is being evaluated as an alternate selection for the tungsten coating if assembled thermocouple tests continue to damage the elements, as mentioned earlier. Prior investigations indicate that tantalum carbide is stable in a hydrogen atmosphere.⁷ Depending on the imperviousness to hydrogen of the carbide structure, which is composed of combinations of TaC and Ta₂C phases,¹¹ one would expect it to adequately protect the tantalum base from hydriding.

In order to compare the effectiveness of each method, parameters for carburizing the tantalum had to be established first. The carburization depth was set at 0.003 inch total for all carbide phases. Samples of tantalum tubes closed at both ends were placed in lampblack inside a resistance furnace and heated to 2000°C for various times. The atmosphere was argon gas. Figure 15 illustrates the resultant total carbide thicknesses and microstructures as a function of time at carburization temperature. As shown for the 2- and 3-hour runs, massive carbides penetrated the wall thickness of each to greater depths than the nominal carbide layer measured. Heat-up and cool-down times were 1 1/2 and 12 hours, respectively. The results of the study indicate that 2 1/2 hours at 2000°C in lampblack and argon will carburize the tantalum surface to the required depth. The tantalum tubes are considerably embrittled by the surface carburization treatment, but not to the extent that handling of the

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sheaths for assembly will result in thermocouple failures. For a nominal 0.003-inch layer, the overall wall thickness increased 0.001 inch. For different types of fixturing to do various lengths, new parameters must be determined. At the present time, parameters for doing 42- to 48-inch long tubes are being determined in a large vertical graphite furnace capable of doing the full length at one time.

After the procedure for carburizing tantalum was established, a program was planned to qualitatively determine which means of protection, tungsten or carbide, is more effective in the prevention of hydrogen embrittlement of the tantalum sheaths. Samples of bare tantalum, vapor-deposited tungsten-coated tantalum, and surface carburized tantalum tubes, each two inches long and closed at both ends, were subjected to flowing hydrogen at ambient pressure and 900° C (1650° F) for times of 1, 2, 4, and 6 1/2 hours. This temperature represents the middle of the most severe hydriding range for tantalum.

When the test time elapsed for each sample, it was carefully removed from the furnace with a pair of tongs, dimensioned, and metallographically examined. During removal, the bare tantalum specimens tended to crumple due to embrittlement, as expected. Post-test dimensions indicate that the wall thickness increased 22% in one hour as a result of the growth from hydriding. This is evident when Figures 16 and 17 are compared. The hydrided region, which appears as a gray phase in the latter photo, and this swelling do not appear in any of the tungsten-coated or surface-carburized samples. The structures shown in Figures 19 and 21 appear relatively unchanged from the control samples of Figures 18 and 20, respectively. However, the surface carburized tubes were more fragile than the tungsten-coated ones when removed from the furnace, partially due to the brittleness of the carbide and, perhaps, partially due to slight hydrogen embrittlement not detectable in the microstructure. None of the tube samples protected by either means show any increase in dimensions for even the 6 1/2-hour test. The overall results of the test are inconclusive as to whether tungsten coating or surface carburization is a more effective preventative of hydriding.

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In order to make the proper selection, another program, now in progress, was designed to measure the hydrogen permeability through various refractory metal combinations. All sheath materials and combinations are being studied, but of particular interest are the tungsten-coated tantalum, surface-carburized tantalum, and the bare tube specimens. Also, a vapor-deposited tungsten tube with a 0.004-inch wall will be included to simulate the tungsten coating alone. This program should yield more decisive results because measurements are being made of the quantities of hydrogen that diffuse through the specimen walls. The samples are 12-inch long tubes closed at one end that will be induction-heated to various temperatures from ambient to 2450°C. Only ambient pressure will be studied at first, with higher pressure work to be done later.

Another program that will be carried out is a study of carbon diffusion rates through the protective layers. This is important since the thermocouple sheaths in service are in direct contact with graphite and the tantalum carburizes quite severely. Two specific considerations will be examined: first is the effect of the vapor-deposited columnar structure as opposed to a wrought structure; second is the effect of service exposure on high-temperature carbon environments on the surface-carburized structure. Until all of these points are investigated, a recommendation of one protection mechanism cannot be made. The primary reason for the present use of tungsten is the availability of a procedure for coating full length tubes (42 inches and over).

V PREVENTION OF METAL-CARBON EUTECTICS BY TUNGSTEN

In addition to the use of coatings to prevent hydriding of tantalum sheaths, tungsten vapor-deposited coatings are used to prevent other refractory metal sheaths from being in direct contact with carbon, thus permitting a metal-carbon eutectic formation and melting. Present designs that utilize molybdenum tubes are limited by reactor operation to a temperature of 4450°R (2210°C), which is also the carbon-molybdenum eutectic temperature. Therefore, the tubes are coated with tungsten by vapor deposition to prevent contact with and hinder the diffusion of carbon. Also, tungsten-26% rhenium tubes have been proposed as an alternate

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selection for sheathing to replace the tantalum on the high-temperature thermocouple. Although this choice would eliminate the need to protect against hydriding, protection is needed against carbon because the tungsten-rhenium alloy forms a eutectic at 2450°C (4900°R),^{8, 9} which is just under the maximum temperature of operation for this thermocouple. Therefore, a tungsten coating is necessary in this case to serve the same purpose as it does for molybdenum in the lower temperature couples.

The value of this coating to protect against metal-carbon eutectic formations when the metals operate in carbon environments at or slightly above the normal eutectic temperatures has never been assessed. For this reason a test program has been planned to subject coated tube samples to the conditions of temperature and time in carbon which are questionable. The specimens are tungsten-coated molybdenum and tungsten-coated tungsten-26% rhenium tubes, each two inches in length and open at both ends. Two thicknesses of tungsten will be studied-- 0.004 inch and 0.008 inch, nominally. The short tubes will be mounted inside thick-walled graphite tubes and heated inductively to 2210°C and 2450°C for Mo and W-26% Re, respectively. Also, experiments will be conducted above these temperatures to determine if the tungsten provides protection beyond the eutectic points. In the case of molybdenum, melting has occurred as low as 2060°C in the presence of carbon on the outside and beryllium oxide on the inside of a tube.¹⁰ The present program may be expanded to make similar determinations on W-26% Re. All samples will be metallographically examined after test for any signs of melting or metal-carbon reactions.

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VI CONCLUSIONS

1. Experimental data indicate that both a vapor-deposited tungsten coating and a surface-carburization treatment protect tantalum from hydrogen embrittlement and dimensional growth.
2. Although one vendor has shown the capability of producing adequate vapor-deposited tungsten coatings, additional process development and control are necessary to achieve consistently reliable coatings.
3. Further development and evaluation are needed to establish the required process control for surface-carburization of various lengths of tantalum tubes.
4. Surface-carburization of tantalum tubing increases the wall thickness and embrittles the material.
5. The vapor-deposited tungsten coating applied to tantalum by Vendor B is superior in quality to that of Vendor A.
6. The feasibility of a vapor-deposited tungsten coating preventing carbon eutectic melting of molybdenum and tungsten-26% rhenium sheaths must be determined experimentally.

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VII RECOMMENDATIONS

1. To assure high-quality coatings for protection of refractory metal sheaths from hydrogen and carbon damage, the following experimental evaluations should be conducted:
 - a. Study of the effectiveness of coatings as a deterrent to metal-carbon eutectic formations and catastrophic melting.
 - b. Additional studies to determine the relative values of a vapor-deposited tungsten coating and a carburized surface layer on tantalum to prevent hydriding.
 - c. Determination of the rates of hydrogen penetration through protective layers as a function of temperature and pressure in order to select a coating that will prevent hydriding.
 - d. Process development and control evaluations on the vapor deposition process to achieve consistently reliable coatings.
2. To achieve a thermocouple capable of measuring temperature to 5000°R (2500°C) in a carbon-hydrogen environment for one hour, a materials and processes program of development and evaluation should be conducted including the following:
 - a. Post-test evaluation of environmentally tested high-temperature thermocouples.
 - b. Determination of chemical compatibility between electrical insulators and refractory metal sheaths or wires.
 - c. Measurement of rates of carburization in refractory metals as a function of temperature.
 - d. Determination of the effects of carbon and hydrogen on the properties or structure of potential electrical insulators.
 - e. Selection and evaluation of high-temperature electrical insulators.
 - f. Determination of effects of nuclear irradiation on individual thermocouple materials.

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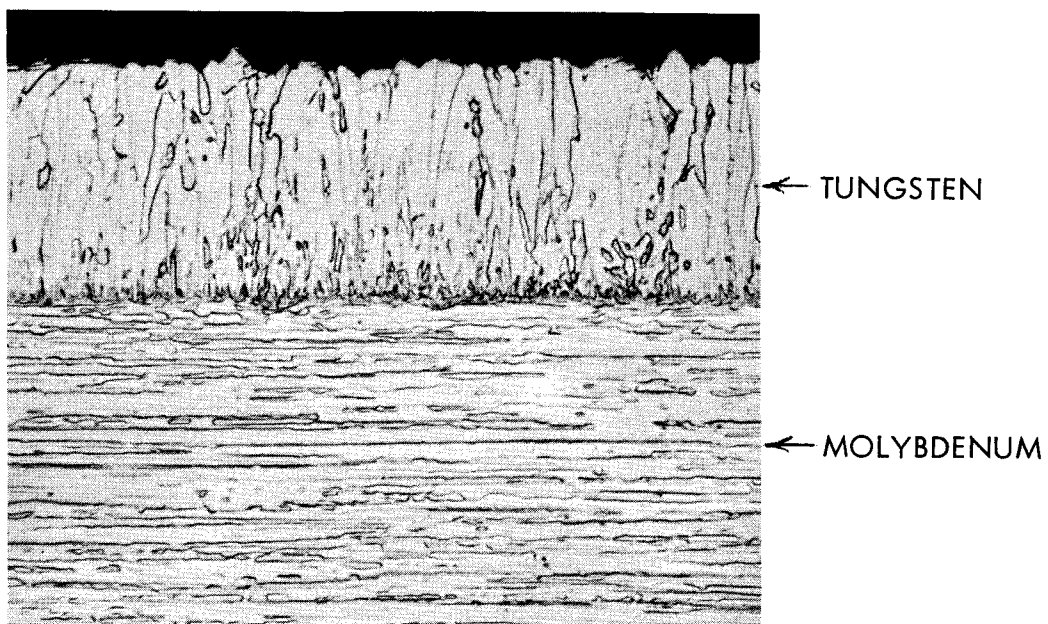


FIGURE 1 500X
TUNGSTEN VAPOR DEPOSITED ON MOLYBDENUM

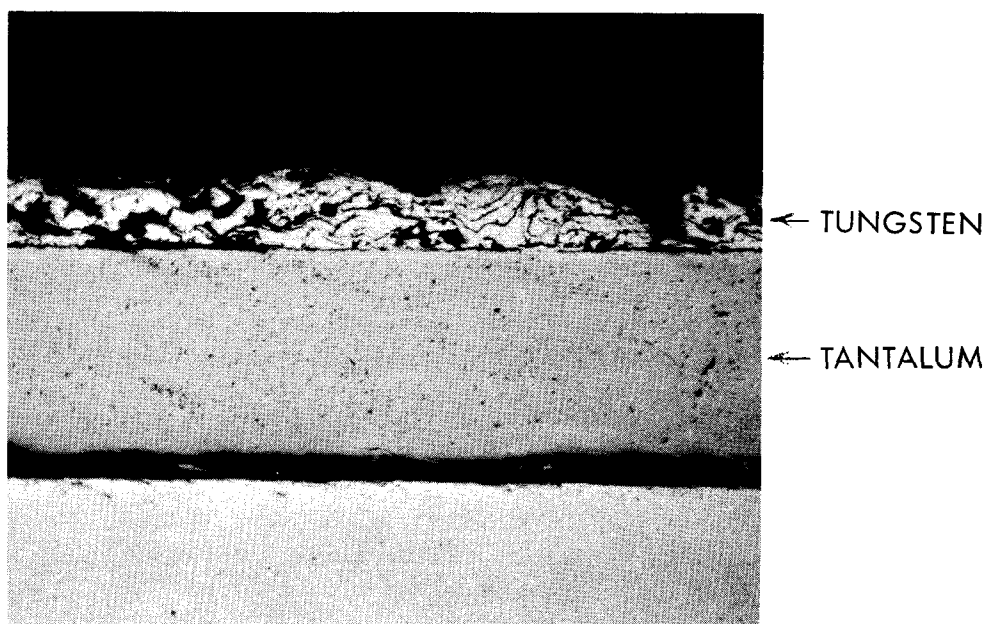


FIGURE 2 150X
TUNGSTEN FLAME SPRAYED ON TANTALUM

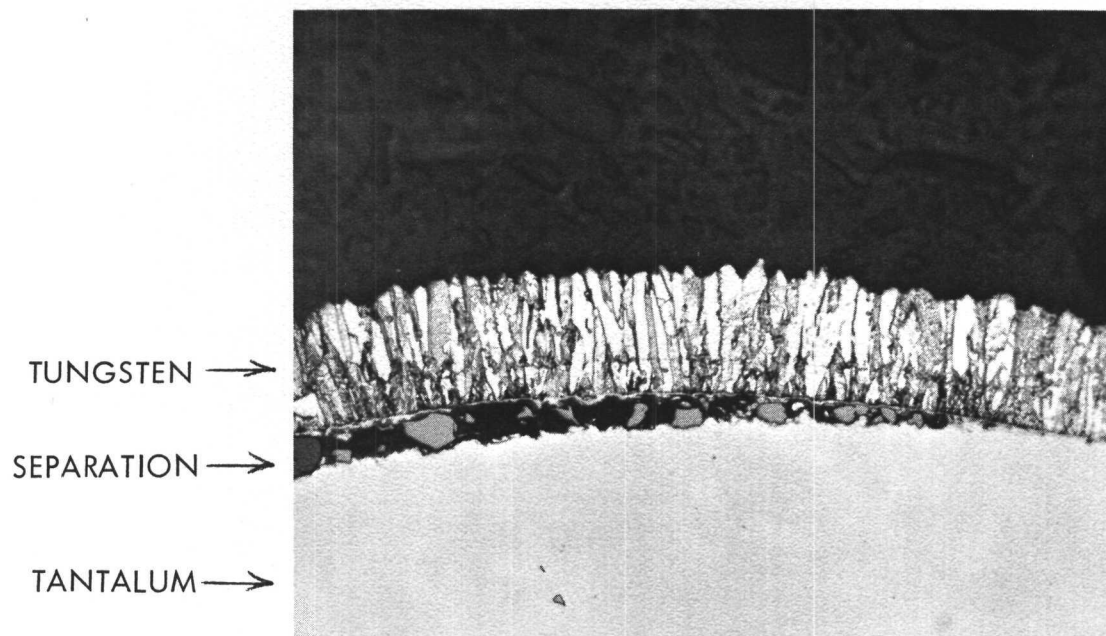


FIGURE 3

250X

SEPARATION BETWEEN TUNGSTEN AND TANTALUM

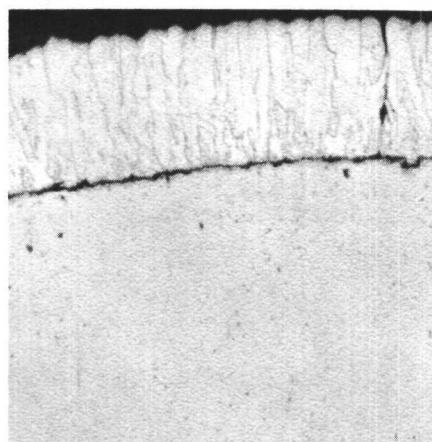


FIGURE 4

200X

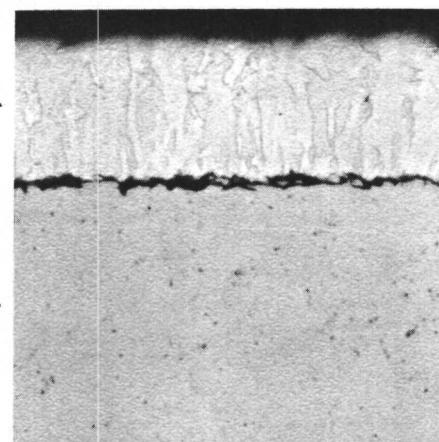


FIGURE 5

400X

VAPOR-DEPOSITED TUNGSTEN ON TANTALUM

VENDOR A

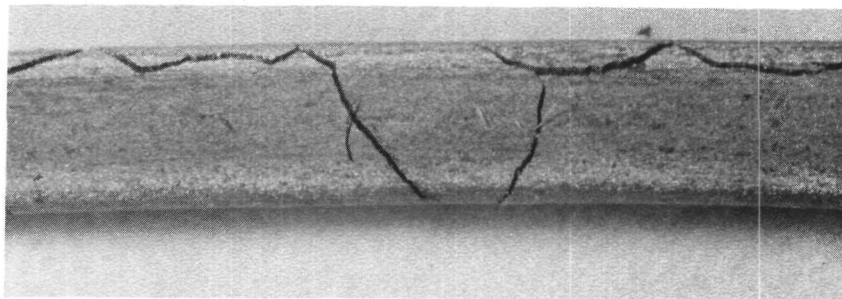
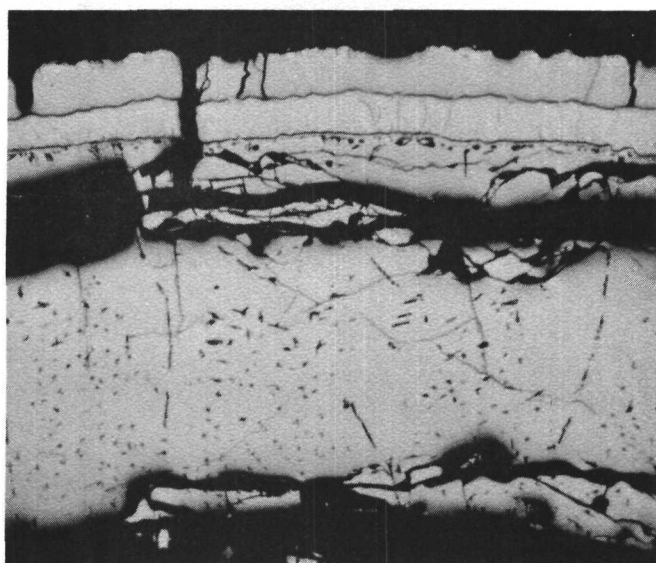


FIGURE 6

10X

TUNGSTEN ON TANTALUM



← CARBURIZED TUNGSTEN

← UNCARBURIZED TUNGSTEN

← HYDRIDED TANTALUM

FIGURE 7

200X

POST-TEST DAMAGE TO TUNGSTEN-COATED TANTALUM SHEATH

COATINGS BY VENDOR A

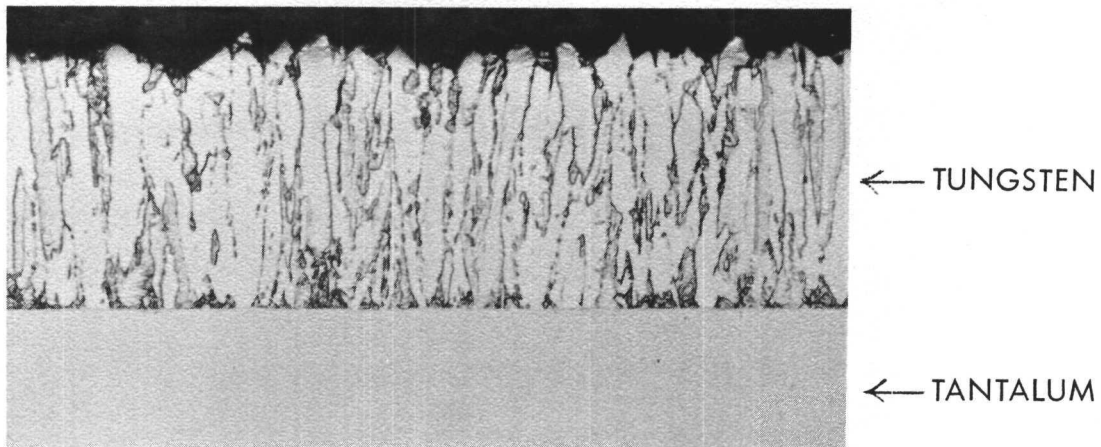


FIGURE 8

400X

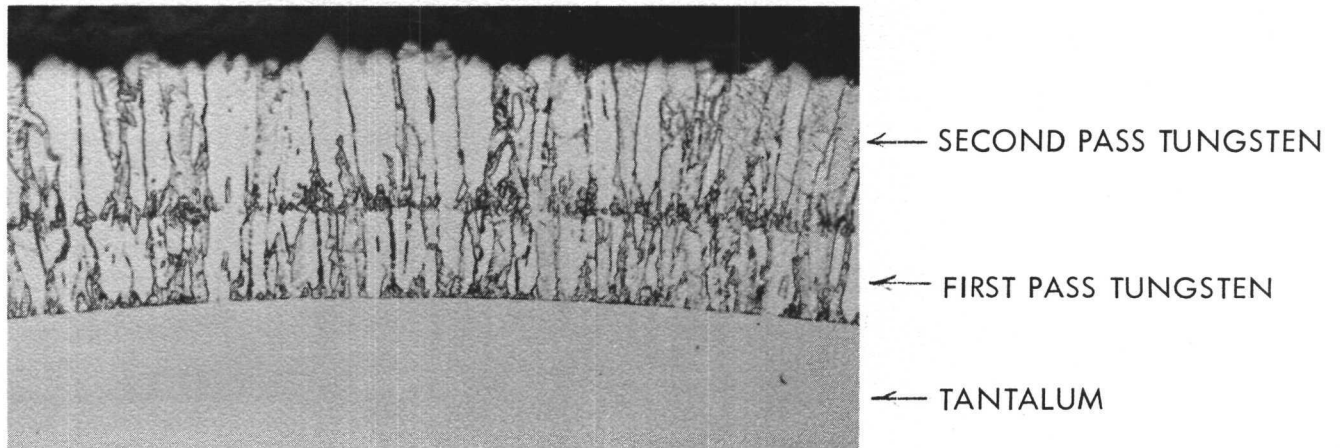


FIGURE 9

400X

TUNGSTEN VAPOR DEPOSITED ON TANTALUM

VENDOR B

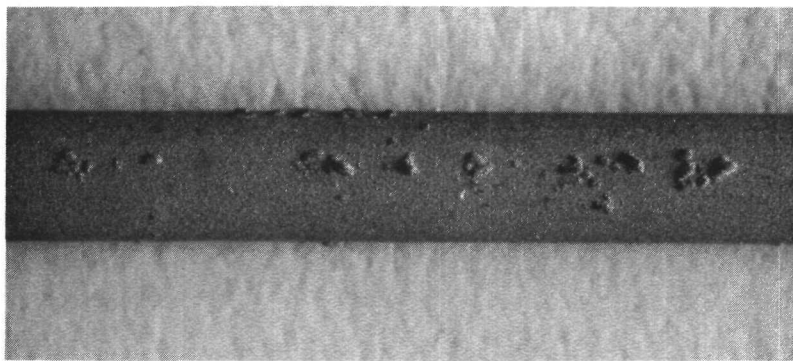


FIGURE 10

10X

NODULAR GROWTH IN TUNGSTEN

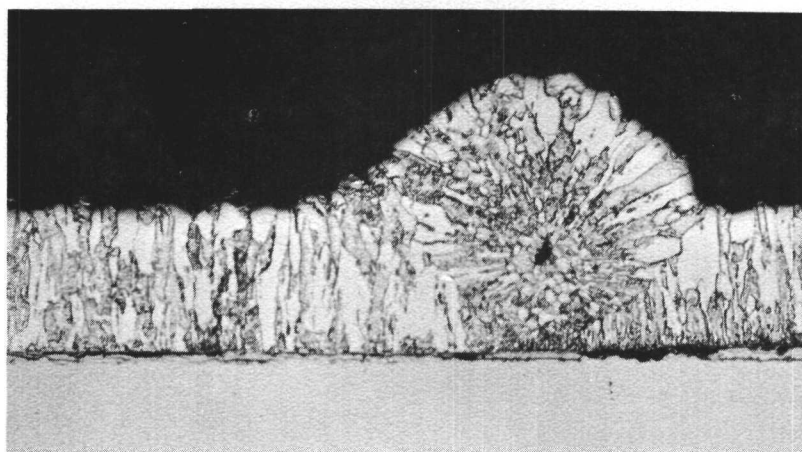


FIGURE 11

250X

NODULAR GROWTH IN TUNGSTEN

← TUNGSTEN

← TANTALUM

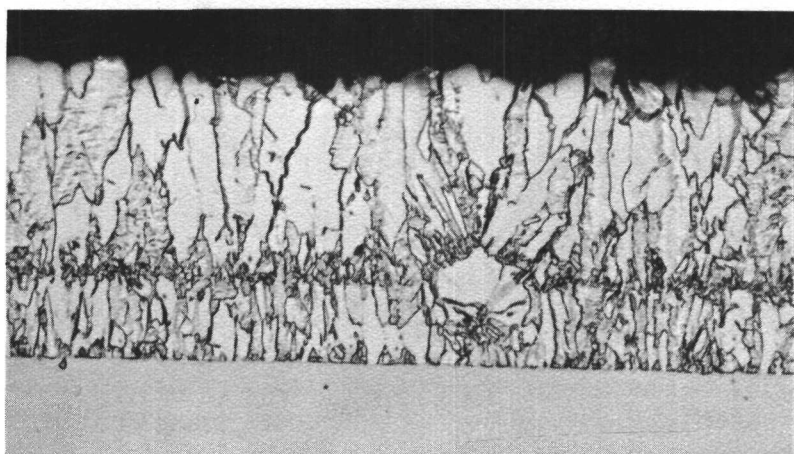


FIGURE 12

400X

START OF NODULE IN TUNGSTEN

← SECOND PASS TUNGSTEN

← FIRST PASS TUNGSTEN

← TANTALUM

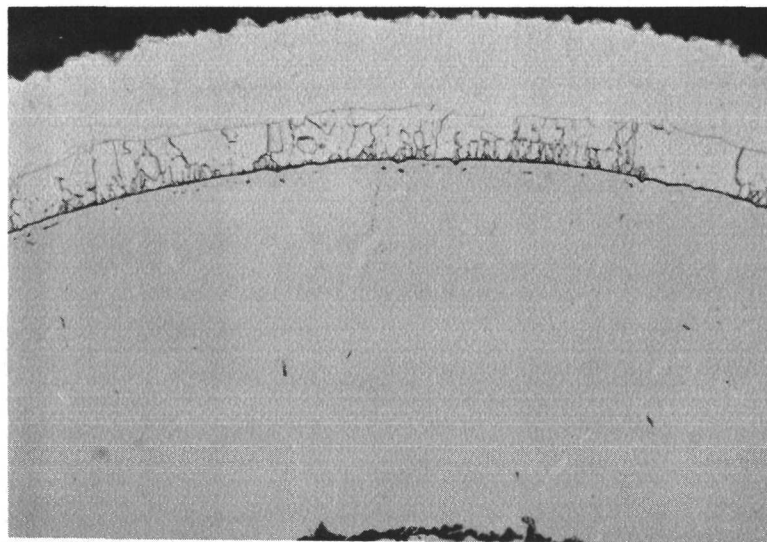


FIGURE 13

200X

VENDOR B

← CARBURIZED TUNGSTEN

← UNCARBURIZED TUNGSTEN

← UNDAMAGED TANTALUM

POST-TEST CONDITION OF
TUNGSTEN-COATED TANTALUM SHEATHS

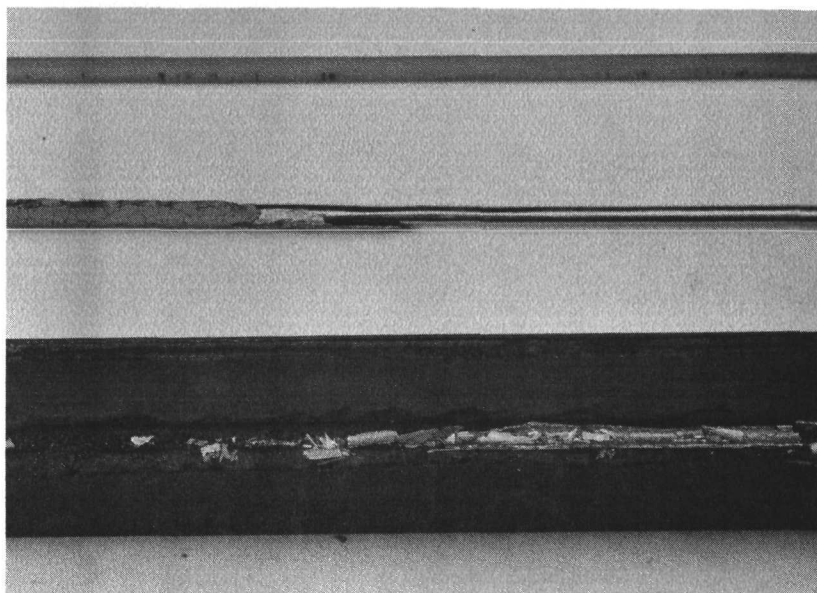


FIGURE 14

1X

← VENDOR B COATED T/C
UNDAMAGED

← VENDOR A COATED T/C
DAMAGED

← ELEMENT WITH REMAINING
PIECES OF TANTALUM AND
TUNGSTEN FROM VENDOR A
COATED T/C

RESULTS OF TANTALUM SURFACE CARBURIZATION

PHOTOS: 250X

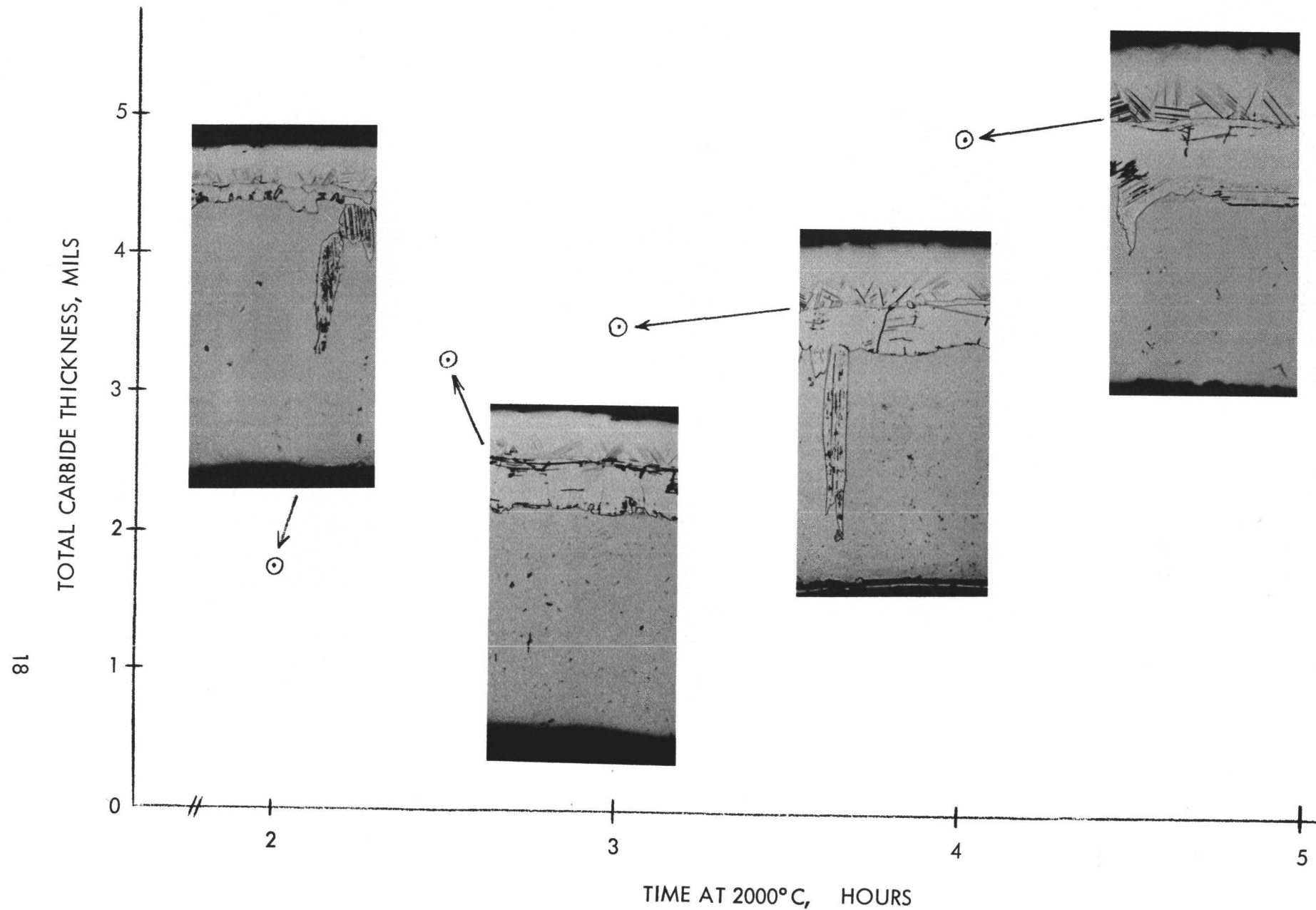


FIGURE 15

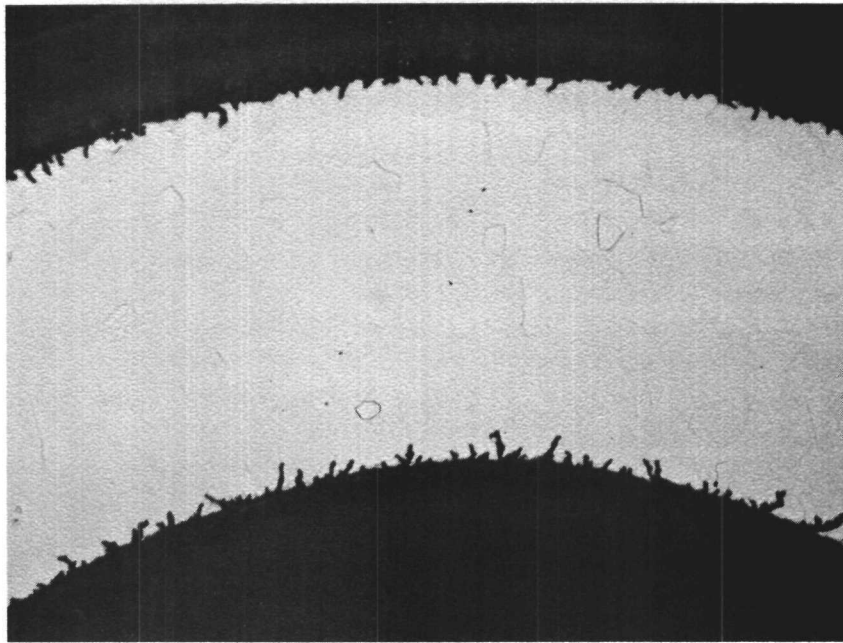
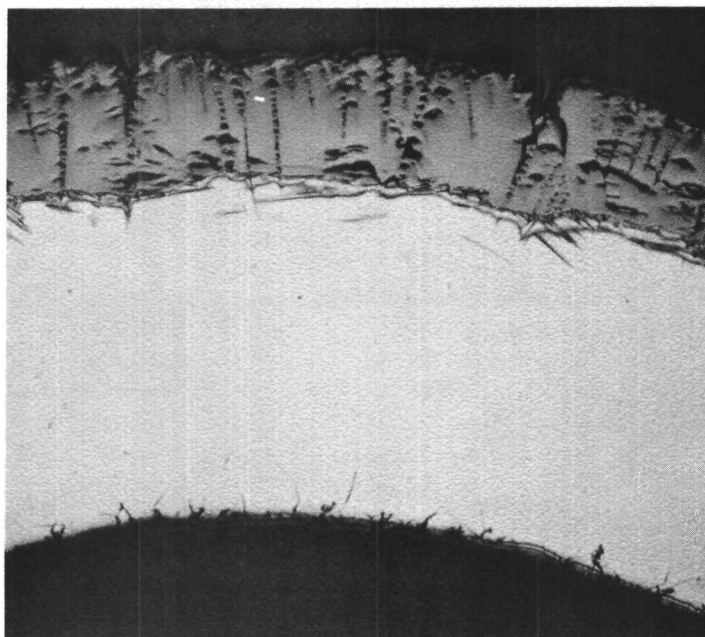


FIGURE 16

PRE-TEST

200X



← TANTALUM HYDRIDE

← TANTALUM

FIGURE 17

POST-TEST

200X

BARE TANTALUM IN HYDROGEN FOR 1 HOUR AT 900°C

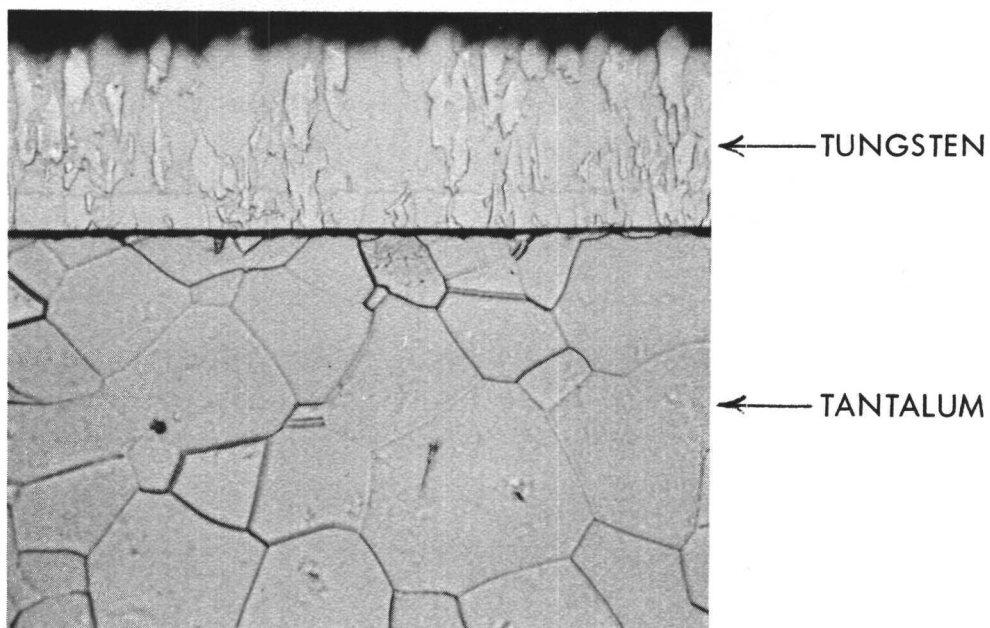


FIGURE 18 PRE-TEST 400X

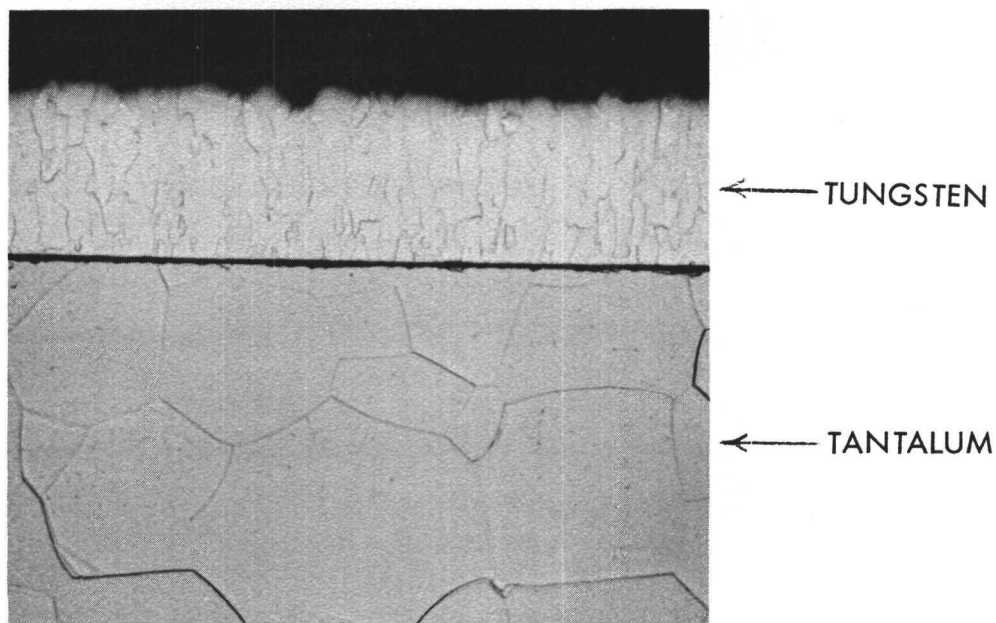
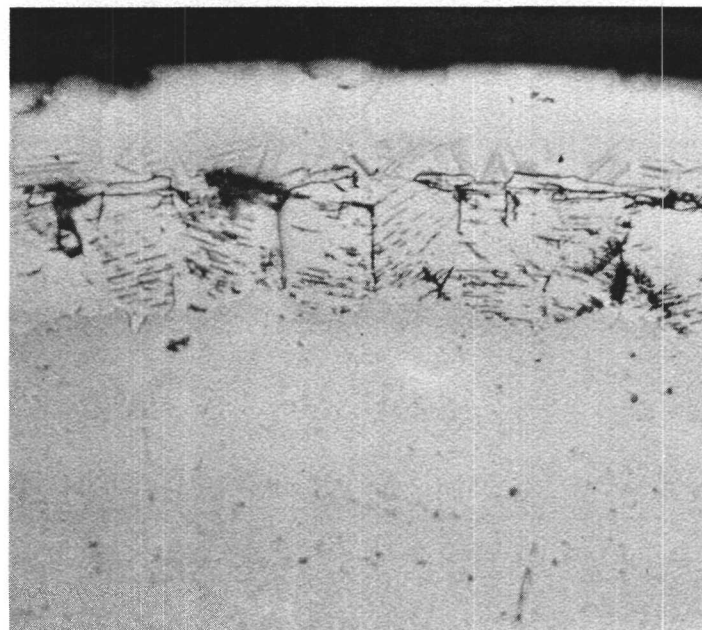


FIGURE 19 POST-TEST 400X

TUNGSTEN-COATED TANTALUM IN
HYDROGEN FOR 1 HOUR AT 900°C

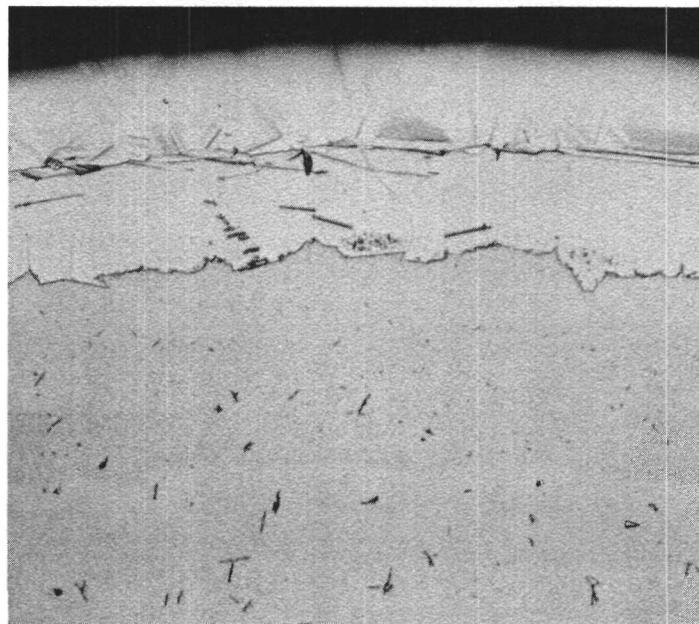


{ CARBURIZED LAYERS
 ← TANTALUM

FIGURE 20

PRE-TEST

400X



{ CARBURIZED LAYERS
 ← TANTALUM

FIGURE 21

POST-TEST

400X

SURFACE-CARBURIZED TANTALUM IN HYDROGEN

FOR 1 HOUR AT 900°C